# WHITING REFINERY EMERGENCY RESPONSE WHITING, LAKE COUNTY, ILLINOIS DATA VALIDATION REPORT

**Date:** April 22, 2014

Laboratory: ALS Environmental (ALS), Holland, Michigan

**Laboratory Project #:** 1404532

Data Validation Performed By: Lisa Graczyk, Weston Solutions, Inc. (WESTON®) Superfund

Technical Assessment and Response Team (START)

**Weston Analytical Work Order #/TDD #**: 20405.016.005.2311.77/ S05-0005-1403-009

This data validation report has been prepared by WESTON START under the START III Region V contract. This report documents the data validation for one soil sample collected for the Whiting Refinery Emergency Response Site that were analyzed for the following parameters and U.S. Environmental Protection Agency methods:

- Total Petroleum Hydrocarbons (TPH) as Oil Range Organics (ORO), Diesel Range Organics (DRO), and Oil Range Organics (ORO) by SW-846 Method 8015
- Dry Bulk Density and Wet Bulk Density by ASTM D5057-90
- Percent Moisture by Standard Method (SM) 2540
- Moisture by Volume by SM 2540

A level II data package was requested from ALS. The data validation was conducted in general accordance with the U.S. EPA "Contract Laboratory Program National Functional Guidance for Superfund Organic Methods Data Review" dated June 2008 and "Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review" dated January 2010. The Attachment contains the results summary sheets with the hand-written qualifiers applied during data validation.

Laboratory Project #: 1404532

#### TPH AS ORO, DRO, AND ORO BY SW-846 METHOD 8015

#### 1. Samples

The following table summarizes the samples for which this data validation is being conducted.

			Date	Date	Date
Samples	Lab ID	Matrix	Collected	Prepared	Analyzed
WCR-Soil01-040814	1404532-01	Soil	4/8/2014	4/11/2014	4/11/2014 -
					4/12/2014

#### 2. <u>Holding Times</u>

The samples were analyzed within holding times for TPH analyses.

#### 3. Blanks

Method blanks were analyzed with the TPH analyses and were free of target compound contamination above the reporting limits.

#### 4. Surrogate Results

The surrogate recoveries were within quality control (QC) limits.

#### 5. Laboratory Control Sample (LCS) Results

The percent recoveries for the LCS results were within the laboratory-established QC limits.

#### 6. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) Results

A site specific MS and MSD were analyzed. The percent recoveries and relative percent differences (RPD) were within QC limits except for as follows. For DRO, the MS recovery was 1.4 percent below the QC limit but the MSD was within QC limits. No qualification was applied for this minor discrepancy.

#### 7. Overall Assessment

The TPH data are acceptable for use based on the information received.

Laboratory Project #: 1404532

# GENERAL CHEMISTRY PARAMETERS (Dry Bulk Density and Wet Bulk Density by ASTM D5057-90, Percent Moisture by SM 2540, and Moisture by Volume by SM 2540)

#### 1. <u>Samples</u>

The following table summarizes the samples for which this data validation is being conducted.

		3.5	Date	
Samples	Lab ID	Matrix	Collected	Date Analyzed
WCR-Soil01-040814	1404532-01	Soil	4/8/2014	4/10/2014 - 4/16/2014

#### 2. <u>Holding Times</u>

The samples were analyzed within an acceptable time frame.

#### 3. Blank Results

A method blank was analyzed with the moisture analysis and moisture was not detected in the blank.

#### 4. LCS Results

The LCS recoveries were within the laboratory-established QC limits.

#### 5. <u>Laboratory Duplicate Results</u>

The laboratory duplicate RPDs were within QC limits.

#### **6.** Overall Assessment

The general chemistry data are acceptable for use based on the information received.

Data Validation Report Whiting Refinery Emergency Response Site ALS Environmental Laboratory Project #: 1404532

#### **ATTACHMENT**

# ALS ENVIRONMENTAL RESULTS SUMMARY



17-Apr-2014

Lisa Graczyk Weston Solutions, Inc 20 North Wacker Drive Suite 1210 Chicago, IL 60606

Re: Whiting Crude Release Work Order: 1404532

Dear Lisa,

ALS Environmental received 1 sample on 09-Apr-2014 for the analyses presented in the following report.

The analytical data provided relates directly to the samples received by ALS Environmental and for only the analyses requested.

Sample results are compliant with NELAP standard requirements and QC results achieved laboratory specifications. Any exceptions are noted in the Case Narrative, or noted with qualifiers in the report or QC batch information. Should this laboratory report need to be reproduced, it should be reproduced in full unless written approval has been obtained from ALS Environmental. Samples will be disposed in 30 days unless storage arrangements are made.

The total number of pages in this report is 12.

If you have any questions regarding this report, please feel free to contact me.

Sincerely,

Electronically approved by: Tom Beamish

Tom Beamish Senior Project Manager ELP ACCREONES

Certificate No: MN 532786

#### **Report of Laboratory Analysis**

ADDRESS 3352 128th Avenue Holland, Michigan 49424-9263 | PHONE (616) 399-6070 | FAX (616) 399-6185 ALS GROUP USA, CORP Part of the ALS Laboratory Group A Campbell Brothers Limited Company

Date: 17-Apr-14

Client: Weston Solutions, Inc

Project: Whiting Crude Release

Work Order: 1404532

**Work Order Sample Summary** 

Lab Samp IDClient Sample IDMatrixTag NumberCollection DateDate ReceivedHold1404532-01WCR-Soil01-040814Soil $04/08/14\ 11:10$  $04/09/14\ 09:30$ 

Date: 17-Apr-14

**Client:** Weston Solutions, Inc **QUALIFIERS, Project:** Whiting Crude Release **ACRONYMS, UNITS** 

WorkOrder: 1404532

Qualifier **Description** Value exceeds Regulatory Limit a Not accredited В Analyte detected in the associated Method Blank above the Reporting Limit E Value above quantitation range Н Analyzed outside of Holding Time Analyte is present at an estimated concentration between the MDL and Report Limit Not offered for accreditation n ND Not Detected at the Reporting Limit Sample amount is > 4 times amount spiked O P Dual Column results percent difference > 40% R RPD above laboratory control limit S Spike Recovery outside laboratory control limits U Analyzed but not detected above the MDL Acronym Description DUP Method Duplicate LCS Laboratory Control Sample LCSD Laboratory Control Sample Duplicate LOD Limit of Detection (see MDL) LOQ Limit of Quantitation (see PQL) MBLK Method Blank MDL Method Detection Limit MS Matrix Spike MSD Matrix Spike Duplicate **PQL** Practical Quantitation Limit RPD Relative Percent Difference TDL Target Detection Limit TNTC Too Numerous To Count A APHA Standard Methods D ASTM

#### SW SW-846 Update III

Е

**Units Reported** 

% of sample Percent of Sample lbs/ft3 Pounds per Cubic Foot

**EPA** 

Milligrams per Kilogram Dry Weight mg/Kg-dry

Description

Date: 17-Apr-14

Client: Weston Solutions, Inc

Project: Whiting Crude Release

**Work Order:** 1404532

**Case Narrative** 

#### QC Comments:

Batch 57444, Method DRO\_8015C\_S, Sample 1404532-01A MS: The matrix spike recovery was outside of the control limit. However, the matrix spike duplicate recovery and the RPD between the MS and MSD were in control. No qualification is required.

 Client:
 Weston Solutions, Inc

 Project:
 Whiting Crude Release
 Work Order: 1404532

 Sample ID:
 WCR-Soil01-040814
 Lab ID: 1404532-01

 Collection Date:
 04/08/14 11:10 AM
 Matrix: SOIL

Analyses	Result	Qual	Report Limit	Units	Dilution Factor	Date Analyzed
DIESEL RANGE ORGANICS BY GC-FID			SW8015	5M	Prep: SW3541 / 4/11/14	Analyst: IT
DRO (C10-C28)	1,100		49	mg/Kg-dry	10	04/12/14 01:54 AM
ORO (C28-C40)	670		49	mg/Kg-dry	10	04/12/14 01:54 AM
Surr: 4-Terphenyl-d14	93.6		39-115	%REC	10	04/12/14 01:54 AM
GASOLINE RANGE ORGANICS BY GC-FI	ID		SW8015	5	Prep: SW5035 / 4/11/14	Analyst: IT
GRO (C6-C10)	57		3.0	mg/Kg-dry	, 1	04/11/14 04:09 PM
Surr: Toluene-d8	108		50-150	%REC	1	04/11/14 04:09 PM
DENSITY - BULK			D5057-9	90		Analyst: MB
Density - Bulk, Dry	68			lbs/ft3	1	04/16/14 02:00 PM
Density - Bulk, Wet	73			lbs/ft3	1	04/16/14 02:00 PM
MOISTURE			A2540 C	3		Analyst: AT
Moisture	16		0.050	% of samp	ole 1	04/10/14 02:36 PM
MOISTURE BY VOLUME			A2540 (	3		Analyst: AT
Moisture, by Volume	8.1			% of samp	ole 1	04/14/14 06:02 PM

**Date:** 17-Apr-14

**Note:** See Qualifiers page for a list of qualifiers and their definitions.

Client: Weston Solutions, Inc

**Work Order:** 1404532

**Project:** Whiting Crude Release

Date: 17-Apr-14

QC BATCH REPORT

Batch ID: <b>57444</b>	Instrument ID GC	8		Metho	d: <b>SW801</b>	5M						
MBLK	Sample ID: DBLKS1-57	7444-5744	4			ι	Jnits: <b>mg/</b>	Kg	Analys	is Date: 04	1/11/14 11	:55 PM
Client ID:		Run IE	): GC8_14	10411A		Se	qNo: <b>271</b> 0	)165	Prep Date: <b>04/1</b>	1/14	DF: 1	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)		ND	4.2									
ORO (C28-C40)		ND	4.2									
Surr: 4-Terphen	yl-d14	1.263	0	1.667		0	75.8	39-115	0			
LCS	Sample ID: DLCSS1-57	444-5744	4			ι	Jnits: <b>mg/</b>	Kg	Analys	is Date: 04	1/12/14 12	:25 PM
Client ID:		Run ID	): GC8_14	10411A		Se	qNo: <b>271</b> (	)168	Prep Date: <b>04/1</b>	1/14	DF: <b>1</b>	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)		157	4.2	166.7		0	94.2	49-124	0			
ORO (C28-C40)		161.3	4.2	166.7		0	96.8	60-130	0			
Surr: 4-Terphen	yl-d14	1.453	0	1.667		0	87.2	39-115	0			
MS	Sample ID: <b>1404532-01</b>	A MS				ι	Jnits: <b>mg/</b>	Kg	Analys	is Date: 04	1/12/14 12	:54 PM
Client ID: WCR-So	oil01-040814	Run ID	): GC8_14	10411A		Se	qNo: <b>271</b> (	)169	Prep Date: <b>04/1</b>	1/14	DF: 10	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)		1120	120	476.6	88	93	47.6	49-130	0			S
ORO (C28-C40)		969.5	120	476.6	561	.7	85.6	60-130	0			
Surr: 4-Terphen	yl-d14	3.489	0	4.766		0	73.2	39-115	0			
MSD	Sample ID: <b>1404532-01</b>	A MSD				ι	Jnits: <b>mg/</b>	Kg	Analys	is Date: 04	1/12/14 01	:24 AN
Client ID: WCR-So	oil01-040814	Run ID	): GC8_14	10411A		Se	qNo: <b>271</b> (	)170	Prep Date: <b>04/1</b>	1/14	DF: <b>10</b>	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
DRO (C10-C28)		1388	120	463.1	89	93	107	49-130	1120	21.4	30	
ORO (C28-C40)		1091	120	463.1	561	.7	114	60-130	969.5	11.8	30	

1404532-01A

The following samples were analyzed in this batch:

QC BATCH REPORT

Client: Weston Solutions, Inc

**Work Order:** 1404532

**Project:** Whiting Crude Release

Batch ID: <b>57453</b>	Instrument ID GC9	Method:	SW8015

MBLK	Sample ID: MBLK-5745	3-57453				ι	Jnits: µg/K	(g	Analy	/sis Date: (	04/11/14 02	:48 PM
Client ID:		Run ID	: GC9_14	0411A		Se	eqNo: <b>270</b> 8	8647	Prep Date: 04	/11/14	DF: <b>1</b>	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
GRO (C6-C10)		ND	2,500									
Surr: Toluene-d8		5222	0	5000		0	104	50-150		0		

LCS	Sample ID: <b>LCS-57453-5</b>	7453				ι	Jnits: <b>µg/K</b>	g	Ana	lysis Date:	04/11/14 02	:21 PM
Client ID:		Run ID:	GC9_14	0411A		Se	qNo: <b>2708</b>	645	Prep Date: 0	4/11/14	DF: <b>1</b>	
Analyte	F	Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
GRO (C6-C10) Surr: Toluene-d8	49	99100 <i>444</i> 2	2,500	500000 <i>5000</i>		0	99.8 88.8	70-130 <i>50-150</i>		0		

MS	Sample ID: 1404555-22A	MS				L	Jnits: <b>µg/K</b>	g	Analy	ysis Date: (	04/11/14 09:	41 PM
Client ID:		Run ID:	GC9_14	0411A		Se	qNo: <b>2710</b>	400	Prep Date: 04	/11/14	DF: 1	
Analyte	F	Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
GRO (C6-C10) Surr: Toluene-d8	44	12600 <i>4364</i>	2,500 0	500000 <i>5000</i>		0	88.5 87.3	70-130 <i>50-150</i>		0		

MSD	Sample ID: 1404555-22A I	MSD				L	Jnits: µg/K	(g	Analys	is Date: (	04/11/14 10:	:07 PM
Client ID:		Run ID:	GC9_14	0411A		Se	qNo: <b>2710</b>	401	Prep Date: <b>04/1</b>	1/14	DF: <b>1</b>	
Analyte	Re	esult	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
GRO (C6-C10) Surr: Toluene-d8		0000 5226	2,500 0	500000 <i>5000</i>		0 0	102 105	70-130 <i>50-150</i>	442600 4364	14. <i>1</i>	2 30 8 30	

The following samples were analyzed in this batch:

1404532-01A

**Client:** Weston Solutions, Inc

**Work Order:** 1404532

**Project:** Whiting Crude Release

QC BATCH REPORT

Batch ID: R138766	Instrument ID MOI	ST		Metho	d: <b>A2540</b>	G						
MBLK	Sample ID: WBLKS-R13	38766				U	nits: <b>% of</b>	sample	Ana	alysis Date: 04	4/10/14 02	2:36 PM
Client ID:		Run II	D: MOIST_	_140410E		Sec	No: <b>270</b> 8	3414	Prep Date:		DF: 1	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture		ND	0.050									
LCS	Sample ID: LCS-R13876	66				U	nits:% of	sample	Ana	alysis Date: 04	4/10/14 02	2:36 PM
Client ID:		Run II	D: MOIST_	_140410E		Sec	No: <b>270</b> 8	3413	Prep Date:		DF: 1	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture		100	0.050	100		0	100	99.5-100.	5	0		
DUP	Sample ID: 1404513-03	A DUP				U	nits:% of	sample	Ana	alysis Date: 04	4/10/14 02	2:36 PM
Client ID:		Run II	D: MOIST	_140410E		Sec	No: <b>270</b> 8	3395	Prep Date:		DF: 1	
						•	•					
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
			PQL 0.050	SPK Val		0	%REC		Value		Limit	Qual
	Sample ID: <b>1404534-02</b> I	Result 12.61				0	0	Limit	Value	%RPD	Limit 20	
Moisture		Result 12.61 B DUP		0		0 U	0	Limit 0-0	Value	%RPD .14 4.12	Limit 20	
Moisture <b>DUP</b>	Sample ID: <b>1404534-02</b>	Result 12.61 B DUP	0.050	0		0 U	0	Limit 0-0	Value 13 Ana	%RPD .14 4.12 alysis Date: <b>0</b> 4	Limit 20	
Moisture  DUP  Client ID:	Sample ID: <b>1404534-02</b>	Result 12.61 B DUP Run II	0.050 D: <b>MOIST</b> _	0 _ <b>140410E</b>	Value SPK Ref	0 U	0 nits: <b>% of</b> qNo: <b>270</b> 8	Limit  0-0  sample 3401  Control	Value  13  Ana Prep Date:  RPD Ref Value	%RPD .14 4.12 alysis Date: <b>0</b> 4	Limit 20 4/10/14 02 DF: 1 RPD Limit	2:36 PM

**Client:** Weston Solutions, Inc

**Work Order:** 1404532

**Project:** Whiting Crude Release

Batch ID: R138992 Instrument ID WETCHEM Method: A2540 G

DUP	Sample ID: 1404532-01	A DUP				U	nits: <b>% of</b>	sample	Analys	is Date: 04	/14/14 06:	02 PM
Client ID: WCR-Soil	01-040814	Run ID:	WETCH	EM_140414	4R	Sec	qNo: <b>271</b> 4	1612	Prep Date:		DF: 1	
Analyte		Result	PQL	SPK Val	SPK Ref Value		%REC	Control Limit	RPD Ref Value	%RPD	RPD Limit	Qual
Moisture, by Volume		9.474	0	0		0	0		8.064	16.1	20	

The following samples were analyzed in this batch:

1404532-01A

QC BATCH REPORT



Cincinnati, OH +1 513 733 5336

Everett, WA +1 425 356 2600 Fort Collins, CO +1 970 490 1511

+1 616 399 6070

Holland, MI

# **Chain of Custody Form**

Houston, TX +1 281 530 5656 Spring City, PA +1 610 948 4903

South Charleston, WV +1 304 356 3168

York, PA +1 717 505 5280

Page \_/ \_of \_ coc ID: 85375

Salt Lake City, UT Middletown, PA +1 717 944 5541 +1 801 266 7700

Enviro	nmental				Al	S Project	Manager:	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1	33		ALS	Work	Order	#: /	14 (	14	532
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Company Name	Weston Solutions, Inc	Bill To Com	pany	Weston	Solutions, I	no									<u>-</u>	<u> </u>		
Send Report To	Lisa Graczyk	Invoice	Attn	Lisa G	raczyk			Du	sry ( Jet	Buc	K D	<del>ZN</del> S	1 1 T Y					
Address	20 North Wacker Drive Suite 1210	Add	dress	20 Nor Sulte 1	th Wacker Dr	ive		E			-		<del></del> :					
City/State/Zip	Chicago, IL 60606	City/Stat	e/Zip		p, jl 60606			G				•						
Phone	(312) 424-3300	P	hone		24-3300			Н										
Fax	(312) 424-3330		Fax	(312) 4	24-3330			1							· 			
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Note: 1. Any changes must be made in writing once samples and COC Form have been submitted to ALS Environmental.
2. Unless otherwise agreed in a formal contract, services provided by ALS Environmental are expressly limited to the terms and conditions stated on the reverse.
3. The Chain of Custody is a legal document. All information must be completed accurately.

Copyright 2011 by ALS Environmental.

From: (219) 405-5409 Pete Wesson **ALS Environmental** 3352 128th Ave

HOLLAND, MI 49424

Origin ID: GRRA

J14101402070326

SHIP TO: (616) 399-6070 Sample Receiving **ALS Environmental 3352 128TH AVE** 

HOLLAND, MI 49424

**BILL SENDER** 

Ship Date: 08APR14 ActWgt: 60.0 LB

CAD: 2264840/INET3490

Delivery Address Bar Code



Dims: 14 X 26 X 15 IN

Ref# Invoice # PO# Dept#

TRK# 0201

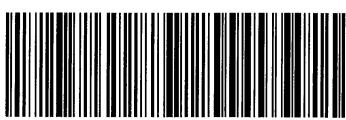
7984 8398 9483

**68 GRRA** 

WED - 09 APR AA STANDARD OVERNIGHT

49424

MI-US GRR



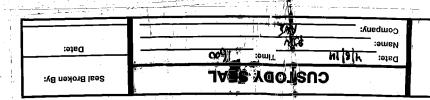


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- 1. Use the 'Print' button on this page to print your label to your laser or inkjet printer.
- 2. Fold the printed page along the horizontal line.
- 3. Place label in shipping pouch and affix it to your shipment so that the barcode portion of the label can be read and scanned.

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Fax. +1 616 399 6185 0409 668 919 I+ 1eT Holland, Michigan 49424 3352 128th Avenue



#### Sample Receipt Checklist

Client Name: WESTON - CHI			Date/Time Received: 09-Apr-14 09:30								
Work Order:	1404532				Received b	y:	KRV	<u>v</u>			
Checklist complement	eted by <u>Siane Sham</u> eSignature <u>Soil</u>	10	0-Apr-14 Date	_	Reviewed by:	Tom Designature					10-Apr-14 Date
Carrier name:	<u>FedEx</u>										
Shipping container/cooler in good condition?		Yes	✓	No 🗆	Not Pi	esent					
Custody seals intact on shipping container/cooler?		Yes	✓	No 🗌	Not Pi	esent					
Custody seals intact on sample bottles?		Yes		No 🗌	Not Pi	esent	✓				
Chain of custody present?		Yes	✓	No 🗌							
Chain of custody signed when relinquished and received?		Yes	✓	No 🗌							
Chain of custody agrees with sample labels?		Yes	✓	No 🗌							
Samples in proper container/bottle?		Yes	✓	No 🗌							
Sample containers intact?			Yes	<b>✓</b>	No 🗌						
Sufficient sample volume for indicated test?			Yes	<b>~</b>	No 🗌						
All samples received within holding time?			Yes	<b>~</b>	No 🗌						
Container/Temp Blank temperature in compliance?			Yes	<b>~</b>	No 🗌						
Sample(s) received on ice? Temperature(s)/Thermometer(s):			Yes 2.4 c	<b>V</b>	No 🗆						
Cooler(s)/Kit(s):											
Date/Time sample(s) sent to storage:				)14 2	2:18:34 PM	No VOA v	iolo oubn	nittod	<b>✓</b>		
Water - VOA vials have zero headspace?			Yes		No □	_		niitea			
Water - pH acceptable upon receipt? pH adjusted? pH adjusted by:			Yes		No 🗌	N/A ✓					
Login Notes:			<u> </u>								
Logiii Notes.											
====	=======	=====			====	===	===			==	====
Client Contacted: Date Contacted:			Person Contacted:								
Contacted By:		Regarding:									
Comments:											
CorrectiveAction	n:								O.F.	OC Do	ao 1 of 1